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Calorimetric Method of Sensitivity Calibration for Far-Infrared Detectors

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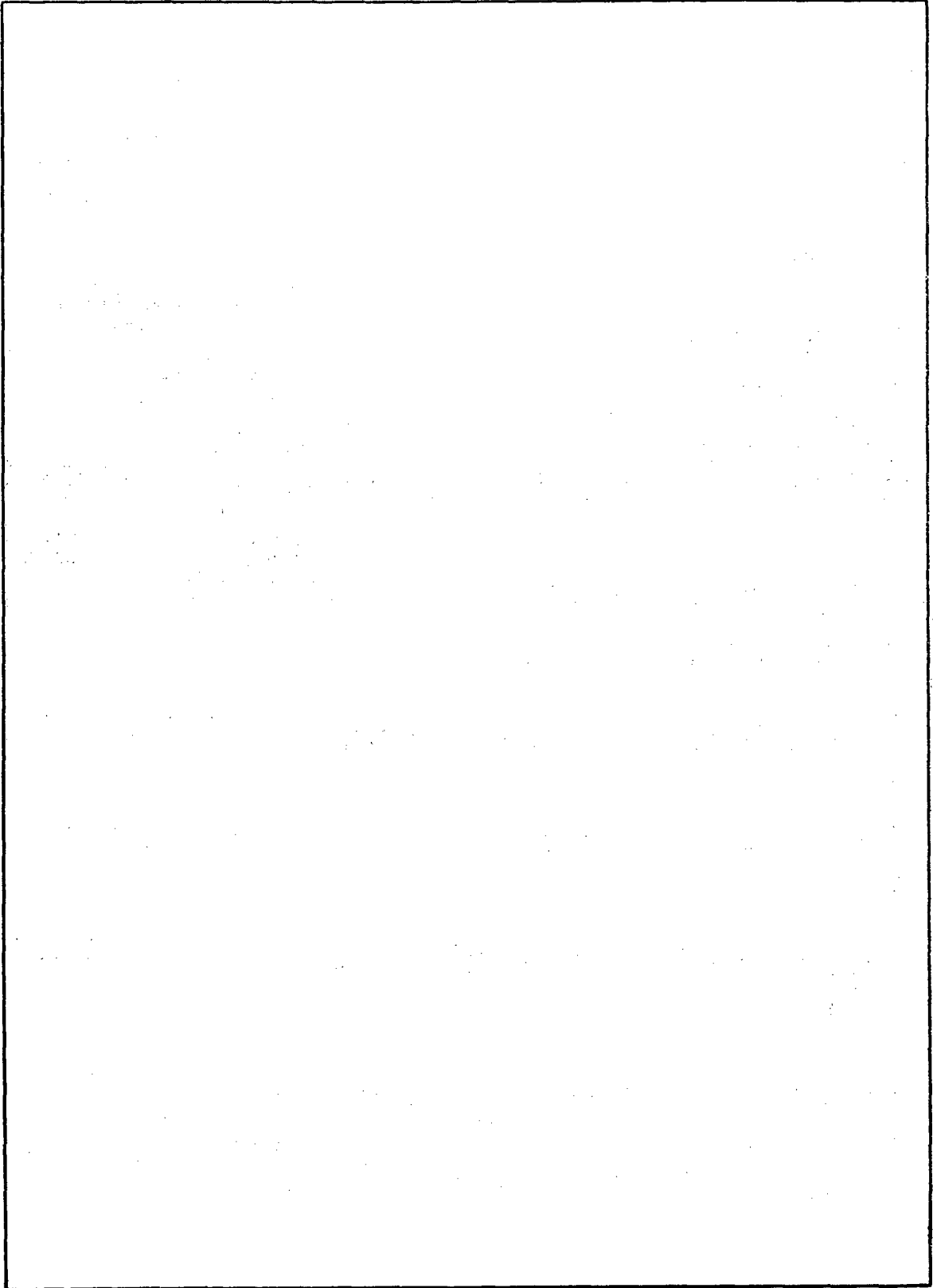
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CALORIMETRIC METHOD OF SENSITIVITY CALIBRATION FOR FAR-INFRARED DETECTORS

INTRODUCTION

The science of far-infrared (defined here as 100 μm to 5 mm) astronomy is relatively new, and its progress depends on continued technological advances. Among these advances are the development of detectors and means of measuring their sensitivities. We report here progress toward a method for calibration of absolute spectral sensitivity in which we observe the detector's response to a monochromatic source whose absolute power output is measured calorimetrically. The calorimeter has been built and can measure powers of less than 1 μW . Preliminary detector sensitivities have been measured, but accurate measurements will be possible only after the completion of a device for rapidly switching the source output between detector and calorimeter.

At present, the most common sensitivity calibration technique is the measurement of the detector's response to a black-body source of known temperature (usually 500-1000K). The response is the black-body emission function multiplied by the detector response, integrated over all wavelengths. Despite its widespread use, however, the technique is open to several objections:

1. The source is assumed to radiate in the far-infrared as a true black-body (that is, its emissivity is unity), a condition very difficult to demonstrate.
2. Accuracy at longer wavelengths is low because most of the detector signal is a response to the much higher black-body emission at the shorter wavelengths.
3. The detector's relative spectral response must be known. Either the detector is assumed to be "flat" (which is rarely true), or the response is measured by comparison with a bolometric detector, which is more likely to have a "flat" response. Even in the latter case, the errors are substantial.

Because of these objections, we have adopted a method that is more direct, although more complicated to carry out. It is simple in concept. A calorimeter is constructed so that it may be illuminated with far-infrared radiation in the same way as the detector to be calibrated. Then the detector and calorimeter responses to various monochromatic sources (klystrons or molecular lasers) are compared. This is obviously not a new method; it extends to shorter wavelengths a technique commonly used at microwave and radio wavelengths.

The major problems are (a) the calorimeter must absorb radiation efficiently, which is difficult, since few surfaces are highly absorbing in the far-infrared; and (b) the calorimeter must be sensitive so that it will measure the low radiation powers that are within

the linear range of the detector and so that weak laser emissions may be used. In addition, the calorimeter must be capable of reasonable accuracy (perhaps 5%) and reasonable speed (a measurement in 1 min is desirable).

The most promising solution appeared to be a calorimeter in the form of an integrating sphere, where a high effective absorption can be obtained even with a surface of moderate absorptance. The calorimeter is cooled to liquid-helium temperature for decreased specific heat and thus increased sensitivity. In addition to its application in detector sensitivity measurements, the calorimeter has also turned out to be of interest to other NRL groups who are developing far-infrared molecular lasers and indium antimonide detectors. This report is concerned mainly with the theory of the calibration technique and with the design and construction of the calorimeter.

DETECTOR CALIBRATION BY THE CALORIMETRIC METHOD

The calibration of a detector is simple in principle. One needs to determine the response of the detector to a known quantity of radiative energy. However, one must consider also how the detector is illuminated, what sort of cavity is being used to increase its effective absorptance, and whether the detector is irradiated by a warm background. In short, calibration of an entire system — the detector plus optics — is necessary. We have chosen as our system a detector mounted in a cavity and fed by a light pipe, with a provision for a cold filter. This configuration is used in several instruments developed by the infrared astronomy group at NRL. The calorimeter may be adapted readily to the same configuration, so that it is illuminated in the same way as the detector, as shown in Fig. 1. The calorimeter measures the absolute amount of radiation emerging from the light pipe, and the detector is calibrated by its response to this amount of radiation. The two quantities thus required for a calibration may be measured simultaneously or, with a beam switch instead of the beamsplitter, in rapid succession.

Figure 1 shows a beamsplitter that divides the source radiation into two parts, so that the two measurements may be made simultaneously. The splitting ratio may be measured by making one pair of measurements with the apparatus as shown and a second pair with the detector and calorimeter interchanged. If the fraction α of the input intensity I emerges through the left-hand port and the fraction β through the right, then in the first set of measurements the calorimeter measures the power $P_1 = \alpha I_1$ and the detector observes $S_1 = \beta I_1$. When the calorimeter and detector are interchanged, the second set of measurements gives $P_2 = \beta I_2$ and $S_2 = \alpha I_2$. Then the splitting ratio is

$$\frac{\alpha}{\beta} = \sqrt{\frac{P_1}{P_2} \frac{S_2}{S_1}}.$$

The beamsplitter need not divide the source intensity into exactly equal parts, although if they differ greatly their ratio may be difficult to measure. The calibration is not affected by absorption or reflection at the beamsplitter, since it is not required that $\alpha + \beta = 1$.

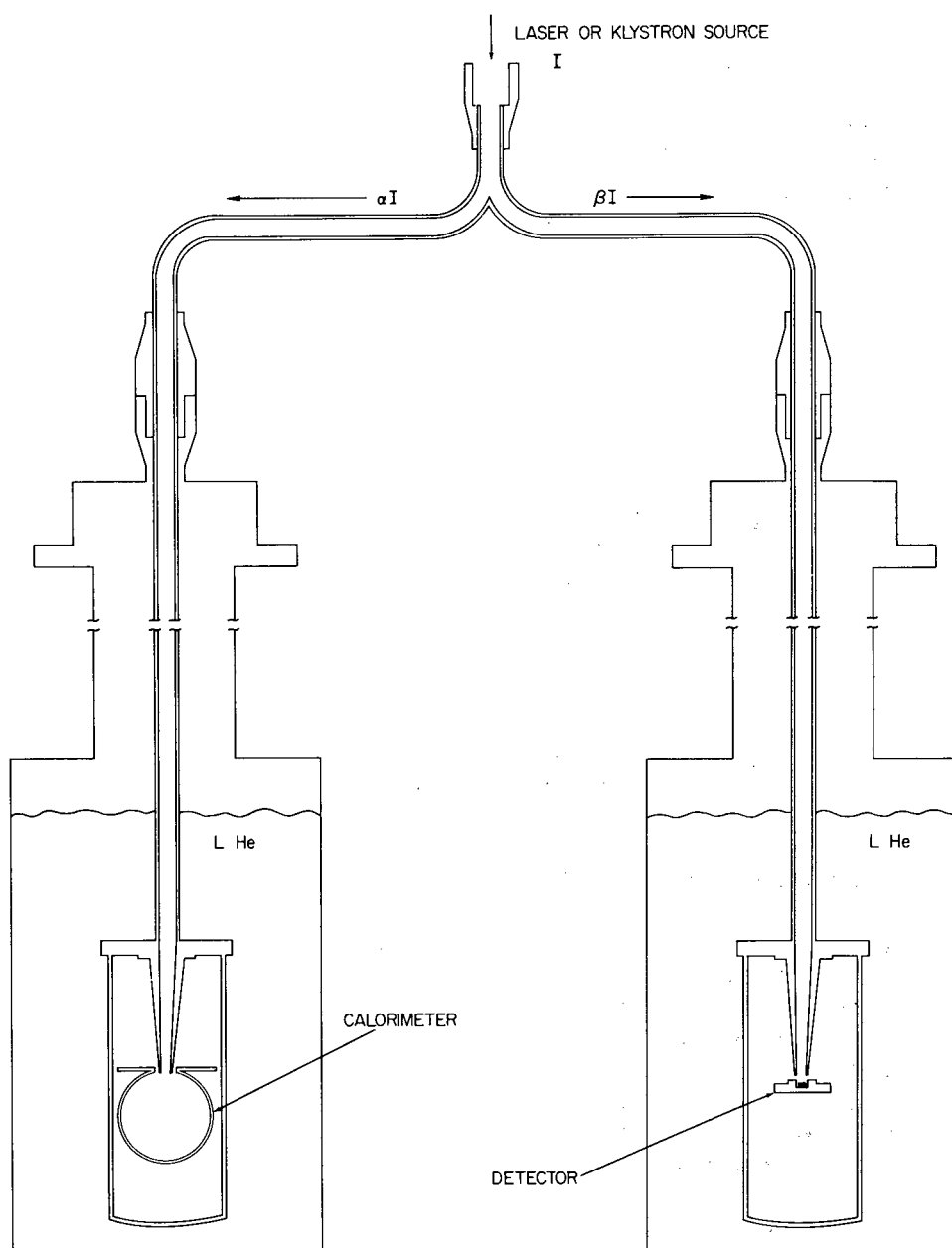


Fig. 1 — Detector calibration system. The beam from a monochromatic source is divided by a beam splitter of known dividing ratio into two beams, one illuminating the detector and the other the calorimeter.

The second method of calibration uses a switch in place of the beamsplitter to direct the source radiation alternately at the detector and the calorimeter. This has the advantage of making available greater power for the calorimeter measurement, but it also requires that the source intensity remain constant during both calorimeter and detector measurements. A sketch of a switch now being constructed is shown in Fig. 2. The switching may be done by a stepping motor or rotary solenoid, and the relative average intensities of the beams may be controlled by leaving the rotating shaft for a longer time in one position than in the other. By switching rapidly — perhaps 10 Hz or faster — one could make an AC calorimetric measurement, which could result in increased sensitivity.

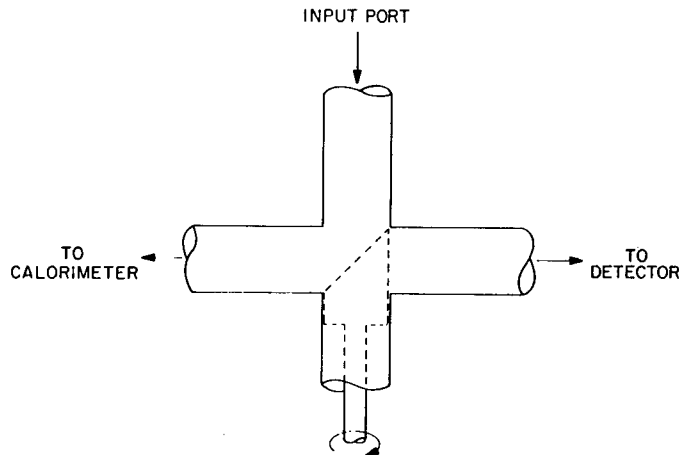


Fig. 2 — Mechanical light-pipe switch to transfer input beam alternately between calorimeter and detector

THEORY OF ISOPERIBOL CALORIMETRY

Our objective is to design an instrument that responds to both radiant and electrical energy, so that the absolute amount of radiant energy can be measured by comparison with a known quantity of electrical energy. Comparison with an electrical source is ideal because the absolute magnitudes of electrical quantities can be measured with an accuracy far exceeding the requirements of the radiant-energy measurement. Considerable theoretical and technological work has recently been done at the National Bureau of Standards [1-4] and elsewhere [5] to develop calorimetry for the measurement of laser radiation, and many of the results are applicable to our problem.

An isoperibol calorimeter is a calorimeter that is in weak thermal contact with a constant-temperature heat sink [1]. Thus, its temperature may differ from that of the sink, but in the absence of a power input (including radiation from a room-temperature window or power dissipated in the temperature measuring resistor), it eventually reaches the sink temperature. In use, only the rate of temperature change is measured, and the equilibrium temperature need never be reached. Other kinds of calorimeters are possible: the calorimeter could be thermally completely isolated, or it could be a phase-change calorimeter, in which the temperature is always constant.

The calorimeter is contained in a housing maintained at the temperature T_s of the thermal sink, and a carefully controlled thermal connection is maintained between the calorimeter and its surroundings. The calorimeter temperature T is measured at some point on the sphere; a change in T is the response to energy put into the calorimeter. Work may be done on the calorimeter by adding radiant energy through the window or electrical energy by means of the resistive heater in the calorimeter. Because of the heat flow while the calorimeter is cooling, not every point of the calorimeter will be at the same temperature, and even after equilibrium is reached there will be thermal gradients if there is an external energy source, such as thermal background radiation entering through the calorimeter window. The equilibrium temperature T_∞ is therefore generally somewhat higher than the sink temperature, but it has been shown [2] that a calibration using the temperature at a single point is accurate in spite of this, as long as the same gradients are established by both electrical and radiant energy sources. We have tried to distribute both radiant and electrical energy over the calorimeter's inner surface, although in fact the possible errors which could be caused by uneven distribution are far too small to affect the required accuracy. Moreover, it is not necessary to measure the absolute magnitude of the temperature; we require only that the measured quantity be linearly proportional to the absolute temperature.

The typical response to work input is illustrated in Fig. 3. When there is no energy input, the temperature falls exponentially toward the equilibrium temperature T_∞ . The exponential temperature decrease is an experimentally verifiable consequence of Newton's law of cooling. When the work period starts, the temperature increases in a way that is difficult (and unnecessary) to predict. When the work input stops, the temperature at first is still difficult to predict, but eventually it again assumes an exponential rate of decrease.

To make an energy measurement, it is necessary to measure (a) the cooling constant of the exponential temperature decrease rate, which is the same both before and after the work input, and (b) the integral under the curve, taken between two points t_i and t_f , both of which must be on the exponential part of the cooling curve. A computerized system for analyzing calorimetry data has been developed by West [4].

The calibration theory is derived from the first law of thermodynamics and Newton's law of cooling. When a thermodynamic system undergoes a transition between two states 1 and 2, the first law relates the amount of work W done on the system to ΔU , the change in internal energy, and to Q , the amount of thermal energy added to the system, in the following way:

$$W = \Delta U - Q.$$

Work is done on the calorimeter by adding either radiant energy or electrical energy. The change in internal energy is $\Delta U = C(T_f - T_i)$, where C is the specific heat, which we assume to be constant. When the calorimeter temperature is higher than that of its surroundings, heat always flows from the calorimeter to the surroundings. The rate of heat flow can be very complicated while work is being done on the calorimeter and for a certain time afterward, but after a suitable length of time the calorimeter cools at a rate such that the rate of temperature change is directly proportional to $T - T_\infty$, an experimentally observable condition known as Newton's law of cooling:

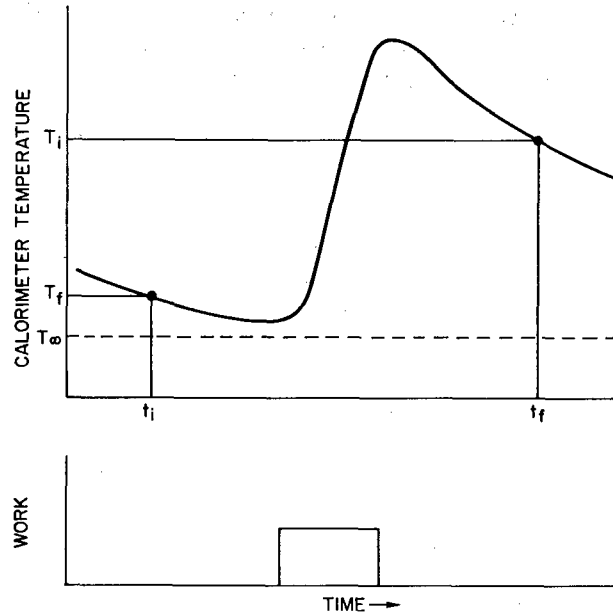


Fig. 3 — Response of an isoperibol calorimeter to an input of work

$$\frac{dT}{dt} = -\epsilon (T - T_{\infty}). \quad (1)$$

The heat that flows during the transition between the two states is

$$\begin{aligned} Q &= C \int_{T_i}^{T_f} dT \\ &= -C\epsilon \int_{t_i}^{t_f} (T - T_{\infty}) dt. \end{aligned}$$

The first law of thermodynamics thus becomes

$$W = C \left[T_f - T_i + \epsilon \int_{t_i}^{t_f} (T - T_{\infty}) dt \right]. \quad (2)$$

This is the essential calorimeter equation. A measurement is made from the data of Fig. 3 by the following procedure:

1. Two times, t_i and t_f , are chosen, one before and one after the work input. The points must fall on the exponentially decreasing part of the cooling curve and must be chosen so that the corresponding temperatures are unequal; that is, $T_i \neq T_f$.

2. The slope dT/dt is measured at times t_i and t_f , and the cooling constant is determined from Newton's law of cooling:

$$\epsilon = \frac{\left(\frac{dT}{dt}\right)_{t_f} - \left(\frac{dT}{dt}\right)_{t_i}}{T_f - T_i}.$$

With ϵ known, Newton's law of cooling gives the equilibrium temperature T_∞ .

3. The adjusted temperature change (the quantity in brackets in Eq. (2)) is determined by calculating the integral $\int_{t_i}^{t_f} (T - T_\infty) dt$.

4. If the work was done by the electrical heater, the specific heat C is then determined (using Eq. (2)) from the known work input W and the adjusted temperature change. If the work was done by an outside source of radiation, its magnitude is calculated from the adjusted temperature change and the specific heat C obtained from a previous or subsequent measurement.

The adjusted temperature rise $T_f - T_i + \epsilon \int_{t_i}^{t_f} (T - T_\infty) dt$ does not depend on which times t_i and t_f are chosen, as long as t_i precedes and t_f follows the work input, and both points fall on an exponentially decreasing part of the cooling curve. For if t_i and t_f are replaced with t_a and t_b , respectively, then Eq. (1) holds within the intervals (t_a, t_i) and (t_f, t_b) , and the new adjusted temperature rise is

$$\begin{aligned} T_b - t_a + \int_{t_a}^{t_b} (T - T_\infty) dt \\ &= T_b - T_a + \epsilon \int_{t_a}^{t_i} (T - T_\infty) dt + \epsilon \int_{t_i}^{t_f} (T - T_\infty) dt + \epsilon \int_{t_f}^{t_b} (T - T_\infty) dt \\ &= T_b - T_a - \int_{t_a}^{t_i} dt + \epsilon \int_{t_i}^{t_f} (T - T_\infty) dt - \int_{t_f}^{t_b} dt \\ &= T_f - T_i + \epsilon \int_{t_i}^{t_f} (T - T_\infty) dt. \end{aligned}$$

DETAILS OF CALORIMETER CONSTRUCTION

Calorimetry is an old science that has recently enjoyed renewed interest because of the need to measure laser output powers. But since these instruments tend to be designed to measure high powers and fast pulses, they are not particularly well suited for low-power measurements. Thus, our high-sensitivity requirement has led to a new design. A cross-section of the calorimeter is shown in Fig. 4, and a photograph in Fig. 5. Some of its numerical dimensions are listed below.

Outer diameter	40 mm
Aperture diameter	7.9 mm
Wall thickness	90 μ m
Copper mass	4.0 g
Total mass, including printed-circuit mounting board	9.6 g
Temperature-sensing resistor:	
20K	68 Ω
4.2K	95 Ω

The calorimeter is a thin, spherical copper shell with a small opening into which radiation is fed by a tapered light pipe. It is contained in a vacuum chamber, one surface of which is in contact with liquid helium and is the cold sink. The calorimeter is thermally isolated by its support (a printed circuit board mounted on three stainless steel posts) but is connected to the cold sink by a wire whose size determines the calorimeter's thermal time constant. A quartz window to complete the vacuum system and a cold black polyethylene filter to reduce room-temperature background radiation are included. Electrical power for absolute calibration is dissipated in a heater with a baseball-seam pattern embedded in the shell.

A spherical shape was chosen for the calorimeter because of the difficulty of obtaining highly absorbing surfaces in the submillimeter radiation range. The effective emissivity ϵ' of the cavity opening is shown from black-body theory [6] to depend only on the ratio a/A (aperture area/sphere area) and the emissivity ϵ of the inside surface. This relationship is given in Fig. 6; it shows that if a/A is small and ϵ is not too small, then the effective emissivity of the cavity opening is close to unity. We don't know the emissivity of the copper shell, but believe it to be greater than 0.05 (corresponding, by Kirchoff's law, to a reflectivity of 0.95), in which case the effective cavity emissivity is greater than 0.85.

The shell is formed by electrodeposition over an aluminum mandrel onto which the printed circuit board has been epoxied. The make-up of the electrochemical solution is as follows:

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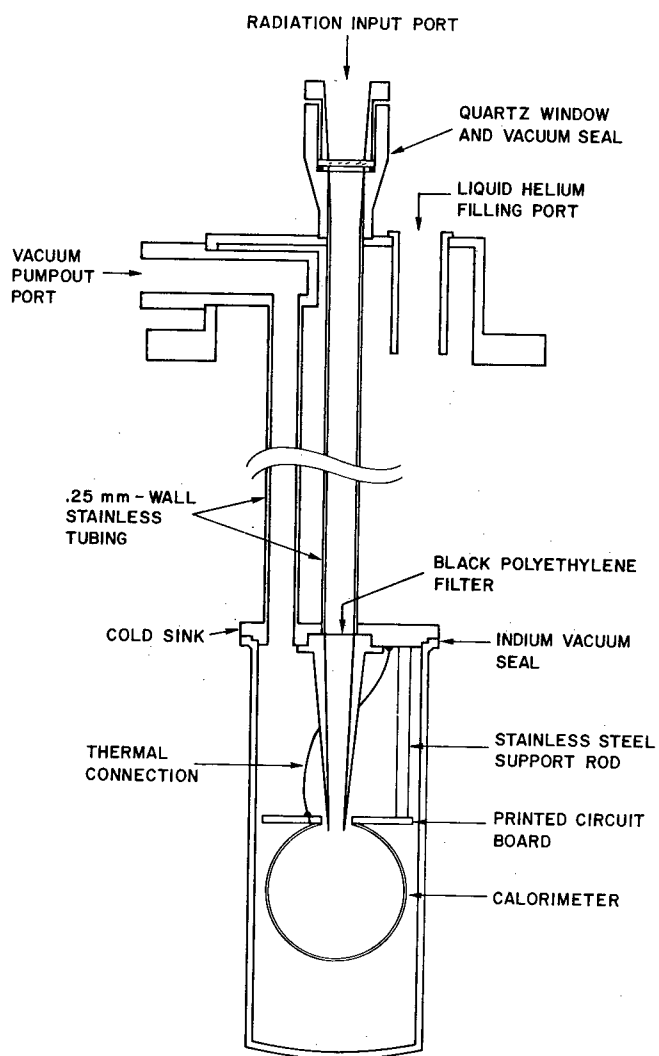
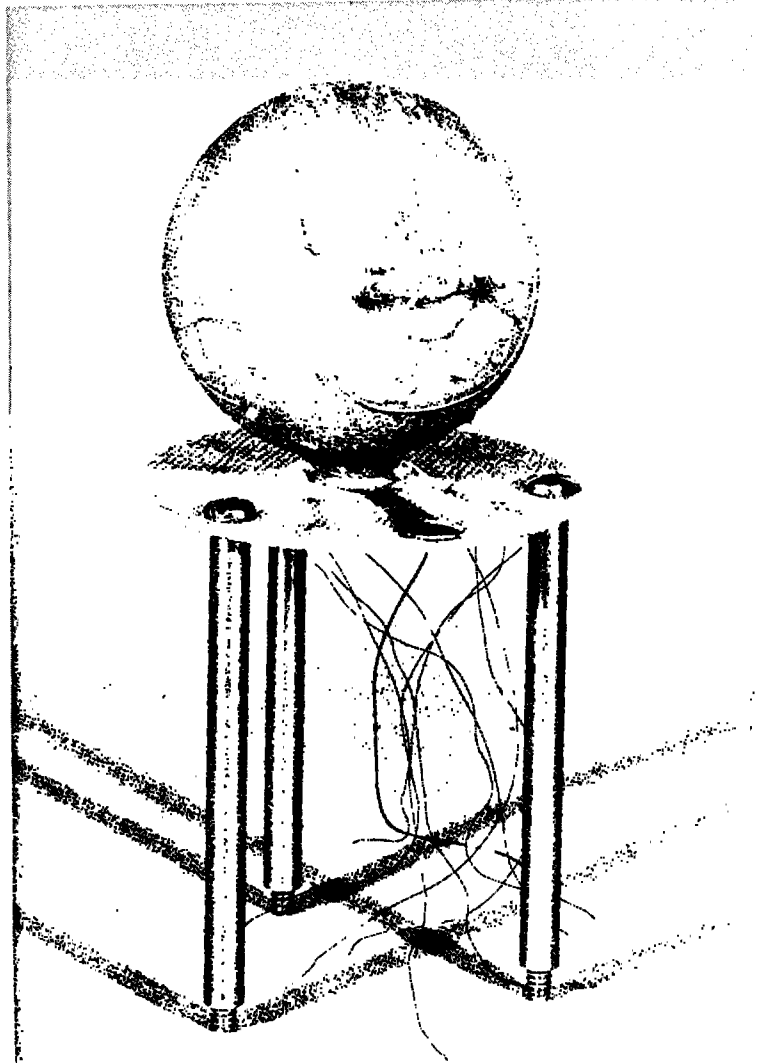


Fig. 4 — Calorimeter cross section

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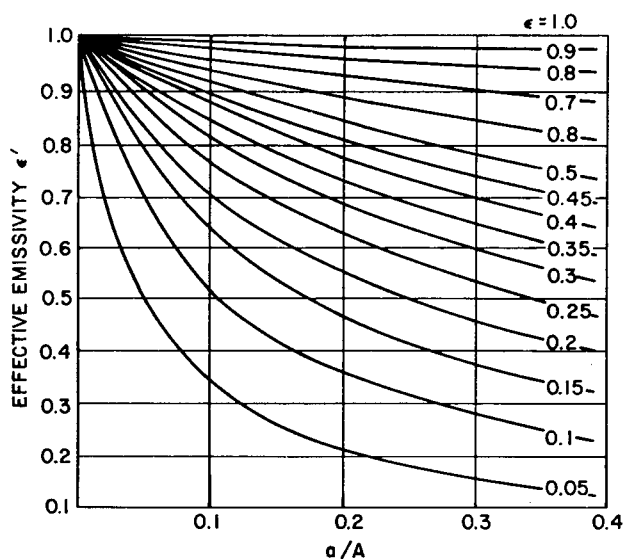


Fig. 6 — Effective emissivity of a spherical cavity as a function of a/A (aperture area/sphere area) and emissivity ϵ of inside surface. From Ref. 6.

Copper sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	750 g
Sulphuric acid, H_2SO_4	225 g
Blackstrap molasses	5 cm ³
Water	31.

The layer of copper is deposited on the mandrel and also over a copper ring on the printed circuit board, thus mechanically bonding the calorimeter sphere to the board. The connection is more rigid if the crack between the copper ring and the mandrel is filled with Aquadag (an electrically conducting emulsion of carbon) before deposition. Electrodeposition is done with an anode completely surrounding the sphere, in an attempt to obtain uniform thickness. Between the printed circuit board and the sphere an additional anode is necessary because of the difficulty of plating "in the crack." Approximately 4 g of copper are deposited during the first deposition. Then the calorimeter is placed in warm NaOH, and the aluminum mandrel is dissolved away, a process requiring about 20 h. The resulting copper shell has walls about 76 μm thick, but it is surprisingly rigid.

The temperature-measuring resistor (an Allen-Bradley old-style ceramic 0.1-W, 68- Ω unit) is then epoxied (with Epoxi-Patch, Type 1C) onto the shell. Two leads are attached to each resistor terminal and tacked down to the sphere with a dab of epoxy; each lead is then fastened down over its whole length with Glyptol, for both mechanical stability and electrical insulation. At the terminal end it is again fastened with epoxy. Thus the leads are in thermal contact with the sphere for a distance of 60 mm, so that the temperature of the resistor is very nearly that of the shell. The heater is deposited in the same way, in the baseball-seam pattern shown in Fig. 5. The wires and resistor are then coated

with electrically conducting Aquadag, and a second copper layer is deposited over the entire sphere, embedding the resistor and heater and their leads in the shell and assuring good thermal contact. This is a painstaking procedure and the most difficult part of the calorimeter manufacture, since there is danger that one of the leads will short to the sphere. The wires are then connected to the printed circuit board, and, finally, connections are made between the printed circuit board and the electronic circuits with fairly robust wires.

OPERATIONAL DETAILS AND QUANTITATIVE RESULTS

The electrical connections to the heater and temperature sensor (Fig. 7) have been made carefully to prevent inaccurate measurements due to the thermal properties of the wires. Thus, the connections between the cold sink electrical feedthroughs and the printed circuit board are made with copper wire to minimize energy dissipation, and the leads to the heater and temperature sensor are doubled so that their resistances, currents, and voltages can be measured by appropriate bridge techniques. The resistance R_T of the temperature sensor is measured with a Kelvin bridge (Keithley 503), so that the measurement is not affected by the resistance of the leads. The maximum power dissipated in this instrument, $0.009 \mu\text{W}$ is too small to affect the calorimeter. Since only small changes in temperature need be measured, the absolute accuracy of the measurement is not important. A convenient way of observing the DC output of the bridge is to use a high-sensitivity oscilloscope preamplifier with a DC offset (Tektronix 1A7).

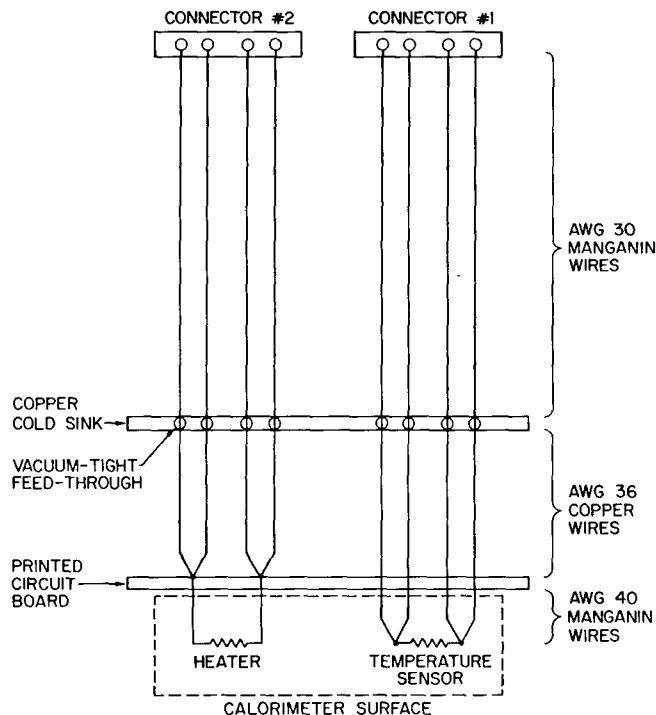


Fig. 7 — Calorimeter electrical connections

The electrical calibrating circuit is very simple. The heater winding is in series with a 1.5-V dry cell and a current-limiting resistor. The power dissipated in the calorimeter is determined from its resistance (22.1Ω , measured with the Kelvin bridge), and from the voltage across it, measured with a high-impedance voltmeter through a pair of leads independent of the current-carrying leads. Most of the heater wire is well heat-sunk to the calorimeter, but part of the energy dissipated in the part of the heater between the shell and the printed circuit board is actually absorbed by the heat sink. We estimate the fraction lost to be about 5% of the total heater energy.

The thermal connection between calorimeter and heat sink is made with a piece of copper wire, which can be easily changed to modify the thermal time constant. We define the thermal time constant as the time required for the calorimeter temperature to rise 90% of the difference between the initial and equilibrium temperatures when a continuous energy input is turned on. At present, the thermal time constant is adjusted to be 30 s.

The initial trials of the calorimeter have been made in a manner somewhat different from that indicated in the theory section. Rather than use a long thermal time constant, we have made the time constant short (30 s) so that the calorimeter comes to thermal equilibrium before and after the calibrating energy is applied. Maximum sensitivity cannot be achieved this way, but the analysis is simpler because no integration is necessary.

The response to an electrical heater power input is shown in Fig. 8. The smallest power to which the calorimeter responded was $0.8 \mu\text{W}$. A crude calibration curve was obtained by measuring the responses to several input powers, as shown in Fig. 9.

A similar response to 3.4-mm klystron radiation was observed (Fig. 10). By comparison with the electrical calibration of Fig. 9 (and application of a 5% correction for imperfect calorimeter absorption), the absolute amount of power emerging from the light pipe was measured to be $23 \mu\text{W}$.

Finally, an InSb detector was calibrated by its response to the known klystron power. The calorimeter was replaced with the detector, and the system was illuminated with 3.4-mm klystron power in the same manner as that used for the klystron power measurement. The klystron power was modulated at a frequency of about 1 kHz. The detector signal was amplified, then rectified and measured with a wave analyzer. This technique is not perfect because the detector measurement and the calibration were made on different days and the source intensity had to be assumed constant. But it made possible a measurement of 120 V/W sensitivity for the detector, which is in agreement with measurements by other means.

SUMMARY AND OUTLINE OF FUTURE DEVELOPMENTS

We have shown the feasibility of measuring the absolute power of small amounts of millimeter-wave radiant energy and of using that energy to calibrate the sensitivity of an InSb detector under conditions similar to those of its astronomical application. However, considerable work must be done to increase the accuracy of the measurements, make

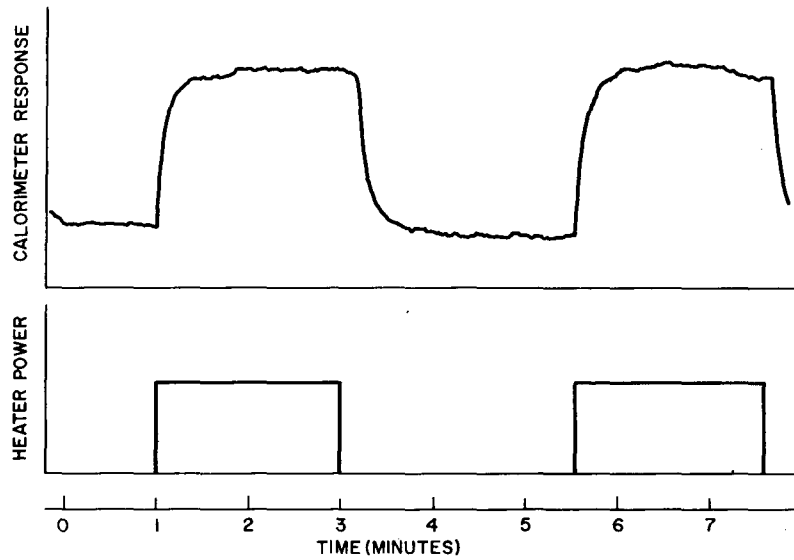


Fig. 8 — Calorimeter response to 20 μW of electrical heater power

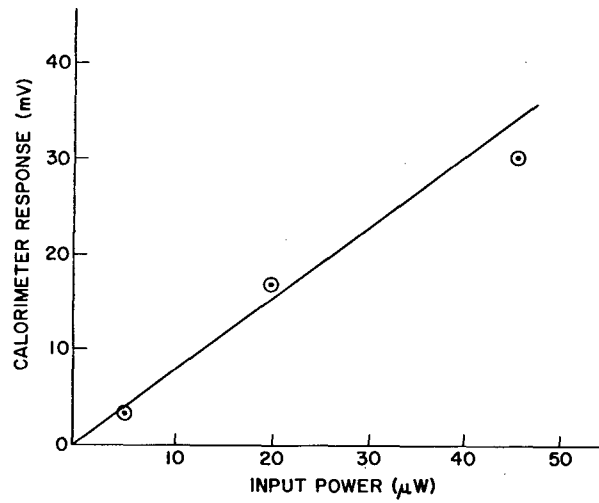


Fig. 9 — Calibration of calorimeter (calorimeter response vs input electrical heater power)

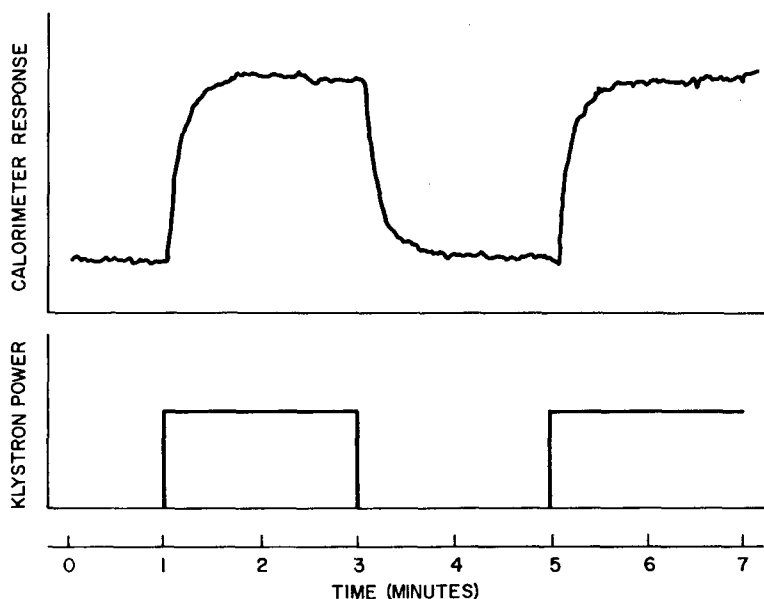


Fig. 10 — Calorimeter response to $23 \mu\text{W}$ of 3.4-mm radiation from a klystron

them more convenient, and find other monochromatic sources that cover a large spectral range. Some of the improvements listed below are already in preparation.

1. A beam switch, built with light pipe components, will allow the beam from the klystron or laser source to be switched rapidly between calorimeter and detector. Then the source intensity need be stable only over periods of minutes.

2. A motor-driven beam switch will make it possible to illuminate calorimeter and detector alternately at a rate of 30 to 100 Hz. Then AC detection techniques may be used for the detector, and source stability requirements will be greatly reduced because detector and calorimeter responses will be measured simultaneously.

3. A computer program for integrating the dT/dt curve will be written so that measurements may be made without requiring the calorimeter to come to thermal equilibrium between data points. The thermal time constant may then be greatly increased, and the accuracy obtainable should be substantially greater.

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